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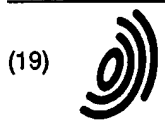
Milk whey protein-containing powder and processed food obtained by using the same

Abstract:

Abstract of EP0821881

Provided is a milk whey protein-containing powder (1) which has characteristic properties of a milk whey protein, such as a gel forming property and the like and (2) which is excellent in a feeling upon eating such as a feeling upon passage through the throat and in taste. This milk whey protein-containing powder is obtained by a process, wherein a solution of a milk whey protein-containing composition is reacted with a transglutaminase, and the reaction solution is heated, and then dried. Data supplied from the esp@cenet database - Worldwide d7b

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(54) **Milk whey protein-containing powder and processed food obtained by using the same**

(57) Provided is a milk whey protein-containing powder

(1) which has characteristic properties of a milk whey protein, such as a gel forming property and the like and

(2) which is excellent in a feeling upon eating such as a feeling upon passage through the throat and in taste. This milk whey protein-containing powder is obtained by a process, wherein a solution of a milk whey protein-containing composition is reacted with a transglutaminase, and the reaction solution is heated, and then dried.

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## Description

Field of the Invention

5 The present invention relates to a milk whey protein-containing powder which is obtained by reacting a solution of a milk whey protein-containing composition with a transglutaminase, then heating the reaction solution at a high temperature, and drying the resulting product, as well as to an animal meat paste food, a fish meat paste food and emulsified food obtained by using the same as a material.

10 Prior Art

Milk whey protein is contained in whey, a by-product obtained in the production of cheese or casein from milk or a skimmed milk powder. In countries where the production of cheese and so forth is conducted prosperously, attempts have been long made to recover milk whey protein and to use the same as a food material as a means for the environmental conservation and the effective use of natural sources.

Further, the production of a milk whey protein concentrate through ultrafiltration has been long reported in which the protein content of the concentrate is between approximately 50 and 70%. Still further, a separate whey protein having a protein content of approximately 90% or more has been produced in order to improve the gelation and emulsifying capacity and the foaming capacity of the protein.

15 Meanwhile, in recent years, studies on a milk whey protein-containing product (for example approximately 30%) having a lower protein content than that of a conventional product as a food material have been actively conducted at a global level in order to effectively utilize the active ingredients of whey, such as protein, sugar, minerals and the like for our diet without processing whey as much as possible from the standpoint of the effective use of natural sources.

When the milk whey protein-containing product which is discharged as a by-product is used in various foods, the foods obtained have a rough feeling as well as an unpleasant feeling upon passage through the throat owing to the milk whey protein. Thus, this is undesirable. The rough feeling and the unpleasant feeling upon passage through the throat are not caused by the overall milk protein, because these problems do not apply to caseins which are a main component of a milk protein.

20 With regard to the problems of the rough feeling and the unpleasant feeling upon passage through the throat which occur in the milk whey protein, a technique by which the molecular weight of this protein is decreased by using an enzyme has been generally employed. However, when a protein is made to have a low molecular weight using an enzyme, the gel forming ability which is one of properties of the milk whey protein is decreased, making it impossible to fully exhibit the properties of the milk whey protein.

Accordingly, a milk whey protein-containing powder (1) which retains the characteristic properties of a milk whey protein, such as gel forming and emulsifying capacity and the like and (2) which provides an excellent feeling upon eating such as a feeling upon passage through the throat and has excellent taste has been in demand. To cope with such problems, the following attempts have been already made.

The retention of the gelation capacity and the decrease of the number of bacteria have been attempted by bringing an non-denatured whey protein powder as such in contact with superheated steam of from 110 to 130°C for from 10 to 20 seconds (refer to Japanese Patent Publication No. 108,191/1995). This method is effective for retaining the gelation capacity and decreasing the number of bacteria. However, the problems such as the rough feeling and the bad feeling upon passage through the throat which are caused by heating even for a short period of time have been unsolved.

A technique has also been studied in which protein particles having an average particle diameter of from approximately 40 to 50  $\mu\text{m}$  are prepared by partially denaturing a milk whey protein (degree of denaturation between 55 and 80%), and added to foods such as mayonnaise, salad sauce, ice cream and the like (refer to world patent application WO 93/25086). However, the problems such as the rough feeling and the bad feeling upon passage through the throat have not completely been eliminated.

Accordingly, the above-mentioned problem of providing a milk whey protein-containing powder which (1) retains the characteristic properties of milk whey protein, such as the gel forming capacity, emulsifying capacity and the like and (2) which has excellent feeling upon eating such as feeling upon passage through the throat and has excellent taste has not yet been realized.

By the way, Shin-Ya Tanimoto and John E. Kinsel (J. Agric. Food Chem., 1988, 36, 281 - 285) have reported on the thermal stability of a polymer mixture obtained by treating  $\beta$ -lactoglobulin with transglutaminase at 99°C or less in view of the protein solubility. Their experiments have revealed indeed that insolubilization of a protein is inhibited. However, the properties of the thus-treated protein, for example, the gel forming and water holding capacity, emulsifying capacity and foaming capacity, have not been examined at all. Of course, the feeling upon eating, such as a feeling upon passage through the throat or the like, has not been referred to at all. Further, this report has described experimental results in a pure system of  $\beta$ -lactoglobulin which is a minor component of a milk protein. There is nothing in this report to sug-

gest the treatment of the overall milk whey protein-containing product that contains various substances.

In a modified milk protein-containing material [Japanese Laid-Open (Kokai) No. 160,957/1991] and a pickle for processed edible meat [Japanese Laid-Open (Kokai) No. 255,426/1995], a milk protein is reacted with a transglutaminase. Casein, a major component of a milk protein, is imagined as this milk protein. These documents do not even suggest the elimination of the problems of the milk whey protein on which the present invention is focused, such as the rough feeling and the bad feeling upon passage through the throat which are not involved in casein.

#### Problems To Be Solved By the Invention

It is an object of the present invention to provide a milk whey protein-containing powder (1) which retains the characteristic properties of a milk whey protein, such as a gel forming and emulsifying capacity and the like and (2) which provides an excellent feeling upon eating such as feeling upon passage through the throat and has excellent taste.

#### Means For Solving the Problems

The present inventors have conducted studies to solve the above-mentioned problems, and have consequently found that a product obtained by reacting a solution of a milk whey protein-containing composition with a transglutaminase, then heating the reaction solution, and drying the resulting product can give an excellent feeling upon passage through the throat without a rough feeling and highly retain properties of milk whey protein such as the gel forming and emulsifying capacity and the like. These findings have led to the completion of the present invention.

That is, the present invention relates to a milk whey protein-containing powder which is obtained by reacting a solution of a milk whey protein-containing composition with a transglutaminase, then heating the reaction mixture, and drying the resulting product, as well as to processed food obtained by using the same.

#### Modes Of Carrying Out the Invention

The present invention is described in detail below.

The solution of the milk whey protein-containing composition in the present invention is typically milk whey containing a milk whey protein which can be obtained as a by-product in the production of cheese, casein or the like from milk or skimmed milk (powder) as a starting material. Such a milk whey is composed mainly of lactose and a protein as solid contents. It is, for example, a solution of a composition comprising approximately 94% of water, approximately 1% of a protein, approximately 4.5% of sugar, approximately 0.5% of ash and a trace amount of fat and the like. Further, a concentrate obtained by concentrating components such as a protein and the like to approximately 2 to 5 times through ultrafiltration, and a milk whey protein-containing solution formed by adding water to a commercially available milk whey protein-containing powder having a protein content of 30% or more are included in the solution of the milk whey protein-containing composition in the present invention.

At any rate, the solution of the milk whey protein-containing composition having a protein content of 10% or less is preferable in view of controlling the interaction between protein molecules in heat sterilization.

The transglutaminase includes a calcium-independent one and a calcium-dependent one. An example of the former is a transglutaminase derived from microorganisms [refer to, for example, Japanese Laid-Open (Kokai) No. 27,471/1989]. Examples of the latter are a transglutaminase derived from guinea pig liver (refer to Japanese Patent Publication (Kouko) No. 50,382/1989), a transglutaminase derived from animal blood (also called Factor XIII), and a transglutaminase derived from fish (refer to, for example, "Journal of Japan Fisheries Academy", by Seki Nobuo et al., vol. 56, No. 1, pp. 125 - 132 (1990)). Further, a transglutaminase produced through gene recombination [refer to Japanese Laid-Open (Kokai) Nos. 300,889/1989, 199,883/1993 and 225,775/1994] is also available. Thus, any of these transglutaminases can be used, and the origin thereof and the method of producing the same are not particularly limited.

In view of the performance and the economics, the calcium-independent transglutaminase is preferable. For example, the transglutaminase derived from microorganisms [Japanese Laid-Open (Kokai) No. 27,471/1989] satisfies all of the above-mentioned conditions, and it is considered most appropriate at present.

An actual method of finally obtaining a desired milk whey protein-containing powder by reacting a solution of a milk whey protein-containing composition with a transglutaminase is specifically described below.

First, a solution of a milk whey protein-containing composition is mixed with a transglutaminase. The amount of the transglutaminase is between 0.2 and 200 units, preferably between 1 and 100 units, more preferably between 5 and 50 units per gram of the protein in the solution of the milk whey protein-containing composition. When the amount of the transglutaminase is less than 0.2 units, the cohesiveness given through the heating is the same as that provided without the addition of the transglutaminase, and the feeling upon eating such as a rough feeling or the like cannot be improved. When it is more than 200 units, the effect provided by the addition of the transglutaminase is the same as that in the

above-mentioned range, which is economically disadvantageous.

In order to react the solution of the milk whey protein-containing composition with the transglutaminase, the solution of the composition containing the transglutaminase is retained under reaction conditions that help to exhibit the enzymatic activity of the transglutaminase. With respect to the reaction conditions of the transglutaminase, the reaction temperature is between approximately 0 and approximately 60°C, and the reaction time is between approximately 5 minutes and approximately 48 hours. The reaction is preferably conducted at a temperature of from approximately 20 to approximately 50°C for from approximately 30 minutes to approximately 2 hours. When the reaction is conducted at quite a low temperature for quite a short period of time, the enzymatic activity is not exhibited. When it is conducted at quite a high temperature for quite a long period of time, the transglutaminase is deactivated.

The solution of the milk whey protein-containing composition resulting from the reaction with the transglutaminase is then heated at from 100 to 140°C for from 1 to 120 seconds for sterilization and deactivation of the enzyme. When the temperature is too high and the time is too long, the protein is thermally denatured at times. This heating is generally UHT, and the treatment is conducted at a high temperature for a short period of time. The UHT may be indirect heating by which to pass steam through a plate or direct heating by which steam is fed directly into a protein solution.

The heat-treated solution of the milk whey protein composition is then dried. With respect to the drying, spray drying is generally appropriate economically. In order to control the thermal denaturation of the protein as much as possible, it is advisable to maintain the exhaust temperature in the dryer at 80°C or less.

The above-mentioned steps make it possible to prepare the milk whey protein-containing powder of the present invention more practically in view of the number of bacteria or the properties of the protein.

The thus-obtained milk whey protein-containing powder can be utilized in various processed foods requiring gel forming capacity, emulsifying capacity and foaming capacity, for example, fish meat paste products such as boiled fish paste product, animal meat paste products such as sausage, milk products such as ice cream and yogurt, and emulsified foods such as mayonnaise and dressing.

The amount of the milk whey protein-containing powder to be added to the processed food is not particularly limited. The ratio thereof to the processed food is between approximately 0.03 and 10%, preferably between approximately 1 and 5%. The thus-obtained processed food has a smooth feeling upon eating without a rough feeling.

The activity unit of the transglutaminase referred to in the present invention is measured and defined as follows. That is, the reaction is conducted using benzyloxycarbonyl-L-glutamylglycine and hydroxylamine as substrates. Hydroxamic acid formed is converted into an iron complex in the presence of trichloroacetic acid. Then, the absorbance of the reaction system is measured at 525 nm. The amount of hydroxamic acid formed is measured using a calibration curve, and the activity is calculated [refer to Japanese Laid-Open (Kokai) Nos. 27,471/1989 and 27,471/1989].

#### Examples

The present invention is illustrated more specifically by referring to the following Examples. However, the technical scope of the present invention is not limited thereto.

#### Example 1

A transglutaminase (specific activity 1 unit/mg) derived from an actinomycetous microorganism belonging to the genus *Streptovorticillium* (*Streptovorticillium mobaraense* IFO 13819) in a predetermined amount shown in Table 1 was added to a solution of a milk whey protein-containing composition obtained by dissolving 100 parts of a milk whey protein concentrate powder (made by DOMO, Netherlands, protein content approximately 35% by weight) in 900 parts of water, and the mixed solution was gently stirred. The pH of the solution of the composition was 6.6. Subsequently, this solution was maintained at 50°C for 30 minutes, and then at 120°C for 10 seconds by blowing high-temperature steam using an ejector-like mixing tube to conduct heating.

The thus heat-treated solution of the composition was injected into a cyclone which had been retained under reduced pressure of 600 mmHg, and rapidly cooled to 60°C. The resulting product was spray-dried at approximately 160°C. In this manner, seven types of the milk whey protein-containing powders shown in Table 1 were obtained. The above-mentioned treatment was conducted except that the transglutaminase was not added, and the resulting product was used as a control.

These products were subjected to an organoleptic evaluation by expert panelists. The results are also shown in Table 1.

Table 1

Amount of TG (units/g • protein)	Hardness (score)	Smoothness (score)	Water holding capacity (%)	Comments
0 (control)	5	5	78.0	rough, brittle, bad upon passage through the throat
0.5	5.6	5.5	82.2	slightly smooth, slightly good upon passage through the throat
1	6.6	6.4	85.0	smooth, good upon passage through the throat
5	7.3	7.1	87.4	very smooth, very good upon passage through the throat
10	7.7	7.9	90.1	very smooth, very good upon passage through the throat
30	8.5	8.1	93.2	very smooth, very good upon passage through the throat
50	9.1	8.3	93.6	smooth, very good upon passage through the throat
100	7.3	6.1	84.4	smooth, good upon passage through the throat
TG: transglutaminase				

As shown in Table 1, the gel of the milk whey protein-containing powder prepared by the addition of the transglutaminase was excellent organoleptically (hardness and smoothness) and physically (water holding capacity) in comparison with the transglutaminase-free product. The gel forming capacity of the milk whey protein-containing powder was evaluated as follows.

(1) Method of preparing a gel:

Thirty grams of water were added to 40 g of the milk whey protein-containing powder, and the mixture was kneaded for 15 minutes using a kneader. This mixture was packed into a casing tube (folding width 47 mm). Then, the resulting product was heated in hot water of 90°C for 40 minutes, and cooled to room temperature with city water to prepare a gel for evaluation.

(2) Evaluation:

The score to evaluate "Hardness" and "Smoothness" in Table 1 is an average of values given by five panelists using a 10-point method in which the control product is given 5 points. Further, the comments are average ones of the five panelists.

"Water holding capacity" was measured using a moisture meter (physicochemical tester manufactured by K.K. Yoshida Seisakusho). That is, a gel having a weight of approximately 4 g was held between two filter papers, and a load of 2 kg was applied thereon for 60 seconds. Then, the water content remaining in the gel was measured from the amount of water absorbed in the filter papers. The water holding capacity was expressed in terms of a ratio (%) of this water content relative to the original water content of the gel. The higher the ratio, the higher the water holding capacity.

Example 2

To a milk whey concentrate (protein content approximately 2%) which was obtained by concentrating milk whey (2%) which was obtained by concentrating milk whey discharged in production of casein through usual ultrafiltration were added 5 units, per gram of the protein in the concentrate, of the same transglutaminase (specific activity 1 unit/mg) as that used in Example 1, and the mixture was gently stirred. This mixture was maintained at 25°C for 60 minutes, and then subjected to indirect steam heating of a UHT plate system at 110°C for 60 seconds. Subsequently, the thus-heat-treated concentrate was subjected to the same procedure as that in Example 1 to obtain a milk whey protein-containing powder (product of this invention).

The above-mentioned treatment was conducted except that the transglutaminase was not added to prepare a milk whey protein-containing powder (control).

Thirty grams of water were added to 40 g of each of the two types of the milk whey protein-containing powders.

The mixture was packed into a casing tube, and heated at 90°C for 40 minutes. The resulting heated gel was subjected to the organoleptic evaluation. Consequently, the inventive product containing the transglutaminase was quite smooth and had an excellent feeling upon passage through the throat in comparison with the control gel.

Example 3

Water (1,500 parts) was added to 100 parts of a separate milk whey protein (made by Nissei Kyoeki K.K., protein content approximately 85% by weight) to obtain a solution of a milk whey protein-containing composition. To this solution were added 10 units, per gram of the protein of this solution, of the same transglutaminase (specific activity 1 unit/mg) as that used in Example 1, and the mixture was gently stirred. The pH of the solution of the composition was 6.6. The solution of the composition was neutralized to a pH of 7.0 with sodium hydroxide, and then maintained at 50°C for 30 minutes. The subsequent procedure was conducted as in Example 1 to obtain a milk whey protein-containing powder (product of this invention).

A milk whey protein-containing powder (control) was prepared in the above-mentioned manner except that a transglutaminase was not added.

To each of the above-mentioned two milk whey protein-containing powders was added water in an amount of two times (by weight) as large as the amount of the powder.

The mixture was packed into a casing tube, and then heated at 90°C for 40 minutes to obtain a heated gel. In the organoleptic evaluation, the gel of the present invention in comparison with the control gel was very smooth, had an excellent feeling upon passage through the throat, and was rich in viscosity.

In the following Examples 4 to 7, various processed foods were produced using the product treated with 10 units, per grams of the protein, of the transglutaminase (TG) as described in Example 1 (milk whey protein-containing powder of the present invention) as well as the TG-free product [TG-free milk whey protein-containing powder (control)] for comparison.

Example 4

Boiled fish paste:

Ground meat of a frozen walleye pollack ("SA-grade Ground Meat" made by Maruha K.K.) was flaked in frozen state.

Thirty grams of sodium chloride and 600 g of ice water were added to 1,000 g of flaked meat, and these were mixed well using a Stefan cutter. Subsequently, to the mixture were added 50 g of wheat starch ("Esusan Ginrei" made by Ajinomoto Co., Inc.), 50 g of sugar, 20 g of mirin (sweet sake), 10 g of a seasoning powder and 30 g of the product treated with 10 units of TG per gram of the protein as described in Example 1. These were mixed using a Stefan cutter such that the temperature of the final product reached 8°C. The thus-obtained paste was packed into a casing tube, warmed at 30°C for 60 minutes, then heated at 90°C for 30 minutes, and cooled to prepare a boiled fish paste casing (product of this invention).

For comparison, a boiled fish paste (control) was prepared in the above-mentioned manner except that 30 g of the product not treated with TG was used instead of 30 g of the product treated with 10 units of TG per gram of the protein.

The two types of the boiled fish pastes were subjected to the organoleptic evaluation, and the results are shown in Table 2 below. As is clear from Table 2, the boiled fish paste (control) containing the product not treated with TG was less elastic, and had a dry feeling upon eating and upon passage through the throat. Meanwhile, the boiled fish paste (inventive product) containing the product treated with 10 units of TG per gram of the protein had a stickiness peculiar to a boiled fish paste, and was smooth and good upon passage through the throat, having a feeling upon eating inherent in a boiled fish paste.

Mechanical tests for a gel strength and a strain were also conducted, and the results are shown in Table 2. In this table, "Gel strength" and "strain" were measured as follows.

(1) Gel strength:

The gel was cut round to a thickness of 30 mm, and a gel strength (g) of the cut piece was measured from a pattern according to a breaking test using a rheometer of Fudo Kogyo K.K. At this time, a ball having a diameter of 7 mm was used as a plunger. The higher the gel strength, the harder the gel.

(2) Strain:

A distance in which the plunger was penetrated through the gel until the gel was broken was measured from the above-mentioned pattern according to the breaking test, and indicated as a strain (mm). The larger the strain, the more flexible the gel.

Table 2

Sample	Gel strength (g)	strain (mm)	Organoleptic evaluation
Invention	556	13.5	good feeling upon passage through the throat without roughness
Control	487	9.7	inappropriate as a fish meat paste with notable roughness

Example 5:

Sausage:

Thirty grams of sodium chloride and 400 g of ice water were added to 1,000 g of pork lean meat and 400 g of pork fat, and these were mixed well using a Stefan cutter.

To the mixture were added 50 g of starch ("Esusan Ginrei" made by Ajinomoto Co., Inc.), 10 g of a seasoning powder and 30 g of the product treated with 10 units of TG per gram of the protein as described in Example 1. These were mixed using a Stefan cutter such that the temperature of the final product reached 10°C. The thus-obtained meat paste was packed into an edible casing tube, then dried in a smoking chamber at 60°C for 30 minutes, smoked at the same temperature for 15 minutes, and finally boiled with steam at 80°C for 40 minutes to prepare a sausage (product of this invention).

For comparison, a sausage (control) was prepared in the above-mentioned manner except that 30 g of the product not treated with TG were used instead of 30 g of the product treated with 10 units of TG per gram of the protein as described in Example 1.

The two types of the sausages were subjected to the organoleptic evaluation. Consequently, the sausage (control) not treated with TG was less elastic, and had a dry feeling upon eating and upon passage through the throat. Meanwhile, the sausage (product of this invention), containing the product treated with 10 units of TG per gram of the protein was so elastic as to give a satisfactory feeling upon biting, was smooth and had a good feeling upon passage through the throat. Thus, it had a feeling upon eating peculiar to a pork sausage.

Example 6

Mayonnaise:

A solution obtained by dissolving 5 g of the product treated with 10 units of TG per gram of the protein as described in Example 1 in 40 g of water was mixed with 20 g of the yolk. Then, 375 g of salad oil, 50 g of vinegar and 10 g of sodium chloride were further added thereto. The emulsification was conducted using a domestic hand mixer to prepare mayonnaise (invention product). For comparison, mayonnaise (control) was prepared in the above-mentioned manner except that 5 g of the product not treated with TG were used instead of 5 g of the product treated with 10 units of TG per gram of the protein as described in Example 1.

The two types of mayonnaise were subjected to the organoleptic evaluation. Consequently, the mayonnaise (control) not treated with TG had a rough feeling upon eating, and quite a bad feeling upon passage through the throat. Meanwhile, the mayonnaise treated with 10 units of TG per gram of the protein was smooth, and had a good feeling upon passage through the throat. It had a feeling upon eating inherent in mayonnaise.

Example 7

## Dressing:

5 The product (3.5 g) treated with 10 units of TG per gram of the protein as described in Example 1 was added to 3 g of water, and the resulting solution was mixed with 14 g of the yolk, 0.75 g of xanthane gum and 0.75 g of guar gum. To this mixed solution were added 205 g of water, 62 g of sugar and 3.5 g of sodium chloride, and these were uniformly mixed. To this aqueous mixed solution were further added 375 g of salad oil, 50 g of vinegar and 10 g of sodium chloride. The emulsification was conducted using a domestic hand mixer to prepare dressing (product of this invention).

10 For comparison, dressing (control) was prepared in the above-mentioned manner except that 3.5 g of the product not treated with TG were used instead of 3.5 g of the product treated with 10 units of TG per gram of the protein as described in Example 1.

The two types of the dressing were subjected to the organoleptic evaluation. Consequently, the dressing (control) not treated with TG had a rough feeling upon eating or an unpleasant strongly acid taste. Meanwhile, the dressing  
15 (product of this invention) treated with 10 units of TG per gram of the protein was smooth, and had a good feeling upon passage through the throat as well as a mild taste. Thus, it had a feeling upon eating and a taste inherent in dressing.

Effects of the Invention

20 The present invention can provide a milk whey protein-containing powder (1) which has characteristic properties of a milk whey protein, such as a gel forming capacity, emulsifying capacity and the like and (2) which is excellent in feeling upon eating such as a feeling upon passage through the throat and in taste. The ability of providing a high molecular weight through  $\epsilon$ -( $\gamma$ -Glu)Lys crosslinking between glutamine and lysine intermolecularly or intramolecularly of a protein with a transglutaminase leads to a dense protein network structure of a milk whey protein.

25 Processed foods prepared by using the milk whey protein-containing powder as a starting material, such as a sausage, a boiled fish paste, dressing and the like, have a smooth feeling, and thus they have quite a good feeling upon eating without roughness.

## Claims

- 30
1. A milk whey protein-containing powder obtainable by reacting a solution of a milk whey protein-containing composition with a transglutaminase, then heating the reaction solution, and drying the resulting product.
  - 35 2. The milk whey protein-containing powder of claim 1, wherein the heating is conducted at from 100 to 140°C.
  3. A process for producing a milk whey protein-containing powder, wherein a solution of a milk whey protein-containing composition is reacted with a transglutaminase, the reaction solution is heated and the resulted product is dried.
  - 40 4. An animal meat paste food, a fish meat paste food or emulsified food which is obtained by using the milk whey protein-containing powder of claim 1 or 2 as a starting material.